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(54) Burner and method for the manufacture of synthetic quartz glass

Brenner und Methode zur Herstellung von synthetischem Quarzglas

Brûleur et procédé de fabrication de verre de silice

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Description

BACKGROUND

Technical Field

[0001] This invention relates to a burner for use in the manufacture of synthetic quartz glass ingots useful as the stock material for excimer laser synthetic quartz glass optical members and large-diameter synthetic quartz glass ingots useful as the stock material for liquid crystal-related large-size photomask substrates. More particularly, it relates to a burner for use in the manufacture of synthetic quartz glass ingots having optical-grade high homogeneity and a minimal change of light transmittance and useful as optical members such as lenses, prisms, mirrors, windows and photomask substrates in excimer laser systems, especially ArF excimer laser systems. The invention also relates to a method for the manufacture of synthetic quartz glass ingots, and to their subsequent processing to make optical members as mentioned above.

Background

[0002] To meet the recent trend of LSI toward higher integration, the photolithography of defining an integrated circuit pattern on a wafer requires an image exposure technique on the order of submicron units. For finer line width patterning, efforts have been made to reduce the wavelength of a light source of the exposure system. In the lithography, a KrF excimer laser (wavelength 248 nm) took over the prior art i-line (wavelength 365 nm) as the mainstream light source in steppers; and the practical use of an ArF excimer laser (wavelength 193 nm) has recently started. Then, the lens for use in steppers is required to have homogeneity, improved UV transmission, and resistance to UV irradiation.

[0003] In order to avoid contamination with metal impurities which cause UV absorption, the synthesis of quartz glass is generally carried out by introducing the vapor of a high purity silicon compound such as silicon tetrachloride directly into an oxyhydrogen flame. Flame hydrolysis takes place to form silica fines, which are directly deposited on a rotating heat-resistant substrate such as quartz glass while being melted and vitrified thereon. In this way, transparent synthetic quartz glass is produced.

[0004] The transparent synthetic quartz glass thus produced exhibits satisfactory light transmittance in the short-wavelength range down to about 190 nm in the LSI field. It has been utilized as a material capable of transmitting UV laser light, specifically i-line and excimer laser light such as KrF (248 nm), XeCl (308 nm), XeBr (282 nm), XeF (351 and 353 nm) and ArF (193 nm), and the four-fold harmonic wave (250 nm) of YAG.

[0005] The absorption of light in the UV region that is newly created by irradiating synthetic quartz glass with UV light having great energy as emitted by an excimer laser is deemed to be due to the paramagnetic defects formed through photo-reaction from intrinsic defects in synthetic quartz glass. Many light absorption bands due to such paramagnetic defects have been identified by ESR spectroscopy, for example, as E' center (Si·) and NBOHC (Si-O·).

[0006] The paramagnetic defects generally have an optical absorption band. When UV light is irradiated to quartz glass, the problematic absorption bands in the UV region due to paramagnetic defects in quartz glass are, for example, at 215 nm due to E' center (Si·) and 260 nm, which has not been accurately identified. These absorption bands are relatively broad and sometimes entail strong absorption. This is a serious problem when quartz glass is used as a transmissive material for ArF and KrF excimer lasers.

[0007] Intrinsic defects in synthetic quartz glass which cause paramagnetic defects arise from structures other than SiO_2 such as Si-OH and Si-Cl and oxygen-depleted or enriched structures such as Si-Si and Si-O-O-Si. As the approach for suppressing paramagnetic defects, it is proposed in JP-A 6-199532 to use a chlorine-free alkoxysilane such as tetramethoxysilane as the silane compound for preventing Si-Cl, one of paramagnetic defects, from being incorporated in glass.

[0008] It is also known that if hydrogen molecules are present in quartz glass in a concentration above a certain level, few defects of E' center (Si·) which are oxygen defects are formed, leading to improved durability to laser damage. Since ArF excimer laser light causes several times serious damages to quartz glass as compared with KrF excimer laser light, the quartz glass for ArF laser application must have several times higher a hydrogen molecule concentration than the quartz glass for KrF laser application.

[0009] It is proposed in JP-A 6-305736 to control the hydrogen molecule concentration in synthetic quartz glass. Depending on the energy using conditions of an ArF laser, the hydrogen molecular concentration in glass is adjusted.

[0010] Now that the efforts to reduce the wavelength of light source have reached excimer laser light having extremely greater energy than the traditional i-line light, active research works have been made on the laser durability of glass.

[0011] Exposure apparatus using such shorter wavelength light include many optical parts such as lenses, windows, prisms, and photomask-forming quartz glass substrates. With respect to projection lens materials among these optical parts used in exposure apparatus, the recent progress is toward a higher NA, the diameter of lens is annually increasing, and the optical homogeneity of lens material is required to be of higher precision. Especially for the ArF excimer laser,

it is required that the initial transmittance of quartz glass, specifically the transmittance at wavelength 193.4 nm over the entire surface of an optical member be close to the theoretical value, the theoretical value at wavelength 193.4 nm being computed to be 99.85% by taking into account multiple reflection. Since the optical system in the exposure apparatus is composed of several to several tens of lenses, it is important that setting an initial transmittance of quartz glass even a little higher restrains the absorption of optical energy within the bulk of quartz glass, thereby minimizing a possibility that the light energy once absorbed is converted to thermal energy to incur a change of density and in some cases, a change of refractive index. In addition to the essential uniformity of refractive index, a reduction of birefringence becomes a crucial problem.

[0012] As stated above, in order to avoid contamination with metal impurities which cause UV absorption, the synthesis of quartz glass is generally carried out by introducing the vapor of a high purity organosilicon compound such as silicon tetrachloride directly into an oxyhydrogen flame. Flame hydrolysis takes place to form silica fines, which are directly deposited on a rotating heat-resistant substrate such as quartz glass and melted and vitrified thereon to form transparent synthetic quartz glass. The synthetic quartz glass ingot thus produced is sliced perpendicular to its growth direction. A distribution of transmittance at wavelength 193.4 nm is determined in a plane of the growth direction. Then, the slice has an in-plane distribution, typically with the tendency that transmittance decreases from the center to the periphery. If the value required for the initial transmittance is at least 99.7% as an internal transmittance, for example, an effective portion of the synthetic quartz glass ingot that can be utilized, generally known as percent yield, is determined by this value.

[0013] Apart from the LSI application, large-size quartz glass substrates for photomasks are now used in the liquid crystal display (LCD) application. It is required to form synthetic quartz glass ingots for use as the stock material therefor to larger diameters, particularly when the percent yield of the manufacturing process of large-size glass substrates is considered. While the mainstream of conventional synthetic quartz glass substrates for IC use is of 6 inch square size, large-size glass substrates have already been required to have one side of 1 meter or longer.

In fabricating large-size quartz glass substrates, as opposed to the synthetic quartz glass ingot for IC use which must have a diameter of about 100 to 140 mm, for example, an ingot which is of a conventional ingot diameter must be increased in length in order to insure a certain product weight. Shaping must be repeated many times until the size is tailored to a desired profile. The situation is detrimental in production yield and efficiency.

[0014] An aspect of the invention is to provide a new burner for use in the manufacture of synthetic quartz glass ingots which serve as the stock material from which synthetic quartz glass members having high optical homogeneity useful as optical parts such as lenses, prisms, windows and photomask-forming quartz glass substrates in excimer laser systems can be readily obtained or synthetic quartz glass members for liquid crystal-related large size glass substrates can be efficiently obtained. Another aspect is to provide a method for the manufacture of synthetic quartz glass ingots using the burner.

[0015] In the manufacture of synthetic quartz glass ingots by vapor phase hydrolysis or oxidative decomposition of a silica-forming compound with the aid of an oxyhydrogen flame, the burner structure for forming a flame is important. EP-A-1 278 009 describes a cylindrical flame shield or flame stabilizer which blows gas such as O_2 or Ar forward around a multi-tube burner, preferably through a honeycomb unit projecting forwardly around the burner. One prior art burner (see EP-A-0 943 586) has a structure including a central triple-tube assembly, a tubular shell surrounding the triple-tube assembly and a plurality of nozzles distributed in a region between the triple-tube assembly and the tubular shell, Our EP-A-1 462 717 (published after the priority date of the present case) describes an additional concentric tubular shell enclosing a further annular series of nozzles. In the embodiment of Fig. 2 of EP-A-1 462 717 a tubular jacket is disposed around the outer tubular shell and at the forward end of the main burner. In the present proposals, replacing the prior art burner by a burner for the manufacture of synthetic quartz glass comprising a main burner comprising at least a central triple-tube assembly, a tubular shell surrounding the triple-tube assembly, and a plurality of nozzles disposed between the triple-tube assembly and the tubular shell and within the confine of the tubular shell, and a double-tube assembly (specified below) disposed around the main burner, we have succeeded in manufacturing synthetic quartz glass ingots from which synthetic quartz glass having high optical homogeneity and synthetic quartz glass members for liquid crystal-related large size glass substrates are obtainable.

[0016] The inventors invention has been to extend the effective portion over the entire region of the synthetic quartz glass ingot. Factors of the manufacturing process that substantially dictate the initial transmittance of a synthetic quartz glass ingot include a burner (structure and set conditions) which is an important constituent of the direct flame process, as well as a starting material or silane compound, a combustible gas (typically hydrogen) and a combustion-supporting gas (typically oxygen) fed thereto, and a balance of these gases. It has been found that the manufacturing process largely depends on the structure of burner among other factors.

[0017] In one aspect, the present invention provides a burner for use in the manufacture of synthetic quartz glass, comprising a main burner having a forwardly-directed front opening and a double-tube assembly surrounding the front opening of the main burner;

a main burner comprising an outer tubular shell surrounding a multi-tube assembly of three or more nested tubes, to define a gas discharge region for feeding a combustible gas in use around the multi-tube assembly and inside the tubular

shell, a plurality of nozzles for feeding combustion-supporting gas in use being distributed in said gas discharge region, and wherein the multi-tube assembly includes a centre tube for feeding a silica-forming compound (silicon-containing compound), a first enclosure tube surrounding the centre tube for feeding a combustion-supporting gas and a second enclosure tube surrounding the first enclosure tube for feeding a combustible gas;

the double-tube assembly being spaced outwardly from the tubular shell of the main burner and comprising an outer tube and an inner tube disposed within the outer tube, defining between said outer and inner tubes a passage for feeding combustion-supporting gas, the outer tube surrounding and projecting forward relative to the front opening of the main burner, whereas the inner tube has its front end axially in register with or behind the front opening of the main burner.

[0018] In a preferred embodiment, the total cross-sectional area of gas discharge ports of the plurality of nozzles disposed in the tubular shell accounts for 5% to 20% of the cross-sectional area of the gas discharge region between the multi-tube assembly and the tubular shell.

[0019] Another aspect of the invention provides a method for the manufacture of a synthetic quartz glass ingot using the burner defined above, comprising the steps of:

placing the burner to face a quartz glass target mounted on a rotating support;

feeding silica-forming compound to the centre tube of the burner, combustion-supporting gas to the first enclosure tube and to the nozzle, combustible gas to the second enclosure tube and to the gas discharge region inside the tubular shell, and combustion-supporting gas to said passage of the double-tube assembly;

forming an oxyhydrogen flame by combustion of the combustible gas in the combustion-supporting gas, thereby subjecting the silica-forming to vapour phase hydrolysis or oxidative decomposition to form silica fines;

depositing the silica fines on the target, with simultaneous melting and vitrification of the deposited silica fines to form quartz glass.

[0020] In a preferred embodiment, the silica-forming compound is a silane or siloxane, the combustion-supporting gas is oxygen, and the combustible gas is hydrogen.

The silica-forming compound and oxygen are fed to the burner such that the molar amount of the silica-forming compound is at least 1.3 times the stoichiometry of oxygen. The molar ratio of the amount of actually fed oxygen to the stoichiometry of oxygen necessary for the silica-forming compound and hydrogen fed to the burner is from 0.6 to 1.3. Typically, the combustion-supporting gas is fed through the double-tube assembly at a flow velocity of 0.5 to 1.3 m/sec.

[0021] Most often, the ingot has a diameter of at least 150 mm. We find that using such a burner, it becomes possible to manufacture synthetic quartz glass ingots which serve as the stock material from which are manufactured synthetic quartz glass members having high optical homogeneity for use in excimer laser systems, especially ArF excimer laser systems, optical members having high laser resistance, and optical members associated with light sources such as excimer lasers, and optical fibers for ultraviolet radiation.

BRIEF DESCRIPTION OF THE DRAWINGS

[0022]

FIG. 1 is a transverse cross-sectional view of a burner for the manufacture of synthetic quartz glass in one embodiment of the invention, gas discharge ports of nozzles being depicted.

FIG. 2 is an elevational view, partially in axial cross section, of the burner of FIG. 1.

FIG. 3 schematically illustrates a prior art burner for the manufacture of synthetic quartz glass, gas discharge ports of nozzles being depicted in cross section.

FIG. 4 schematically illustrates an exemplary synthetic quartz glass manufacturing system.

FIG. 5 is a graph showing the radial distribution of initial transmittance of synthetic quartz glass ingots of Examples and Comparative Examples.

EXPLANATIONS OPTIONS AND PREFERENCES

[0023] A burner for use in the manufacture of synthetic quartz glass ingots comprises a main burner which includes a multi-tube assembly of a three or more tube construction, a tubular shell surrounding the multi-tube assembly, and a plurality of nozzles disposed within the confine of the tubular shell. An outer tube assembly is disposed around the main burner.

[0024] Referring to FIGS. 1 and 2, a burner according to one embodiment is illustrated. A multi-tube assembly 1 (here, triple-tube) has a center tube 2, a first enclosure tube 3 around the center tube to define an (annular) second passage, and a second enclosure tube 4 around the first enclosure tube 3 to define an (annular) third passage. These tubes 2, 3, 4 are concentric or nested. The multi-tube assembly 1 is surrounded at a spacing by a concentric tubular shell 5. Plural individual (non-coaxial, non-nested) nozzles 6 are arrayed in a nozzle region defined between the tubular shell 5 and the triple-tube assembly 1 and within the confine of the tubular shell 5. The multi-tube assembly 1, tubular shell 5

and nozzles 6 constitute together a main burner 7 which has a forward opening (at the top in FIG. 2).

[0025] According our new proposals, an outer, double-tube assembly 8 is disposed so as to surround at least the forward opening of the main burner 7. The double-tube assembly 8 includes an outer tube 9 and an inner tube 10 disposed inside the outer tube 9 and outside the tubular shell 5 of the main burner 7. The forward end of the outer tube 9 of the assembly 8 surrounds the forward opening of the main burner 7 and projects forward thereof. The outer tube 9 provides a guard so that the gas stream from the main burner 7 may not be spread outside the tubular shell 9. The forward end of the inner tube 10 is disposed in register or substantially in register with the forward opening of the main burner 7 although in other embodiments it may be behind it. A first functional aspect of the outer tube assembly is the mentioned forwardly-projecting outer guard tube. A second functional aspect is the gas outlet between its inner and outer elements 9,10, for combustion-supporting gas. This annular outlet can substantially surround the tubular shell 5. Desirably it is substantially in axial register with, or behind, the shell's opening. The outer guard tube 9 projects forwardly past the outlet. The outlet and the inner element 10 of the assembly are radially spaced out from the shell 5.

[0026] It is understood that the burner has an axis, to which tubes and shells extend generally parallel. As used herein, the terms "outside" or "outer" and "inside" or "inner" refer to radial positions with respect to the burner axis. Also, the terms "forward" and "backward" refer to relative positions along the burner axis. In the illustrated embodiment, all tubes and shells are shaped cylindrical and arranged in a concentric fashion to define annular spaces therebetween. However these shapes are not critical.

[0027] Through the center tube 2, a silica-forming compound is fed and channeled, and generally oxygen gas or carrier gas is additionally fed and channeled. Through the second passage (within the confine of the first enclosure tube 3), a combustion-supporting gas such as oxygen is fed and channeled. Through the third passage (within the confine of the second enclosure tube 4), a combustible gas such as hydrogen is fed and channeled. Through the nozzles 6 and the double-tube assembly 8 (between the outer and inner tubes 9 and 10), a combustion-supporting gas such as oxygen is fed and channeled. Through the tubular shell 5, a combustible gas such as hydrogen is fed and channeled to flow about the nozzles 6.

[0028] In a preferred embodiment, the total cross-sectional area of gas discharge ports of the plurality of nozzles 6 disposed in the tubular shell 5, that is, the total cross-sectional area of lumens of nozzles 6, accounts for at least 5%, more preferably 5 to 20%, and most preferably 8 to 13% of the cross-sectional area of a gas discharge region between the triple-tube assembly 1 and the tubular shell 5, that is, the cross-sectional area of an annular space between the assembly 1 and the shell 5 (i.e., the cross-sectional area of an entire annular space between the assembly 1 and the shell 5 provided that the nozzles 6 are omitted).

[0029] The double-tube assembly 8 disposed outside the main burner 7 is typically made of quartz. It is also desirable that the combustion-supporting gas be channeled through the double-tube assembly 8 at a flow velocity of 0.5 to 1.3 m/sec. A flow velocity of less than 0.5 m/sec may allow for undesirable back fire whereas a flow velocity of more than 1.3 m/sec may disturb the flame of the main burner.

[0030] The number of nozzles 6 may be determined in accordance with the above conditions. The flow velocity of the combustion-supporting gas through the double-tube assembly 8 may be determined in accordance with the clearance between the outer and inner tubes 9 and 10 and the desired flow rate of combustion-supporting gas.

[0031] As compared with the prior art burner structure, the provision of the double-tube assembly around the tubular shell of the main burner ensures that when a synthetic quartz glass ingot is produced by the direct flame process, the temperature distribution in the melting face from the center to the periphery of the ingot growing face is more uniform; the high-temperature region at the center is expanded to the periphery. Thus the silica structure is formed under the same conditions between the center and the periphery during the deposition/fusion/vitrification process of silica fines on the ingot melting/growing face. This makes it possible to prevent the initial transmittance of the periphery of an ingot from lowering to below that of the center and to minimize the difference in initial transmittance between the periphery and the center. At the same time, the molten area is spread, making it possible to increase the diameter of a synthetic quartz glass ingot.

[0032] The provision of the double-tube assembly around the tubular shell of the prior art burner structure ensures that in a flame produced by the inventive burner, the high-temperature region is extended from inside flame to outside flame. This outside flame is applied to a peripheral portion of the ingot melting/growing face. The structure allowing the combustion-supporting gas to be channeled between the tubular shell of the main burner and the double-tube assembly ensures to increase the combustion efficiency of a combustible gas such as hydrogen gas fed around the nozzle group, making it possible to extend the high-temperature region throughout the flame. This is particularly true when the proportion of the cross-sectional area of nozzles disposed between the multi-tube assembly and the tubular shell is at least 5%. Further, the enclosure of the forward end of the main burner by the outer tube of the double-tube assembly prevents the flame from being disordered by gas streams within the furnace, concentrating the flame power.

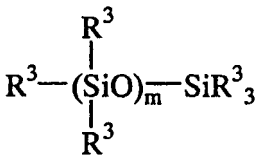
[0033] Now it is described how to produce a synthetic quartz glass ingot using the inventive burner. The burner is placed to face a quartz glass target mounted on a rotating support. A silica-forming compound, a combustible gas such as hydrogen gas, and a combustion-supporting gas such as oxygen gas are separately fed to the tubes of the burner

to form an oxyhydrogen flame with which the compound undergoes vapor phase hydrolysis or oxidative decomposition to form silica fines which deposit on the target. The silica fines are simultaneously melted and vitrified to form a synthetic quartz glass ingot.

[0034] The starting material, silica-forming compound used herein is typically an organosilicon compound which is preferably selected from silane compounds and siloxane compounds represented by the following general formulae (1), (2) and (3).

$$(R^1)_nSi(OR^2)_{4-n} \qquad (1)$$

Herein each of R^1 and R^2, which may be the same or different, is a monovalent aliphatic hydrocarbon group, hydrogen or halogen atom and n is an integer of 0 to 4.



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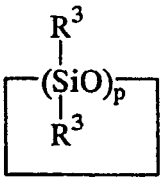


(3)

Herein R^3 is hydrogen or a monovalent aliphatic hydrocarbon group, m is an integer of at least 1, especially 1 or 2, and p is an integer of 3 to 5.

[0035] Examples of the monovalent aliphatic hydrocarbon groups represented by R^1, R^2 and R^3 include alkyl groups of 1 to 4 carbon atoms, such as methyl, ethyl, propyl, n-butyl and tert-butyl, cycloalkyl groups of 3 to 6 carbon atoms such as cyclohexyl, and alkenyl groups of 2 to 4 carbon atoms such as vinyl and allyl.

[0036] Examples of the silane compound represented by formula (1) include $SiCl_4$, CH_3SiCl_3, $Si(OCH_3)_4$, $Si(OCH_2CH_3)_4$, and $CH_3Si(OCH_3)_3$. Examples of the siloxane compounds represented by formulae (2) and (3) include hexamethyldisiloxane, hexamethylcyclotrisiloxane, octamethylcyclotetrasiloxane, and decamethylcyclopentasiloxane.

[0037] The silane or siloxane compound, a combustible gas (e.g., hydrogen, carbon monoxide, methane or propane) and a combustion-sustaining gas (e.g., oxygen) are fed to the burner for forming an oxyhydrogen flame.

[0038] An apparatus for producing a synthetic quartz glass ingot using the inventive burner may be of either vertical or lateral type.

[0039] The synthetic quartz glass ingot produced using the inventive burner preferably has an internal transmittance at wavelength 193.4 nm of at least 99.70%. The reason is that when the ingot is finally used as an optical member, it is sometimes required that the transmittance at the service wavelength which is 193.4 nm in the case of an ArF excimer laser, for example, be at least 99.70% in internal transmittance. If the internal transmittance is less than 99.70%, there is a possibility that when ArF excimer laser light is transmitted by a quartz glass member, light energy is absorbed and converted to thermal energy, which can cause changes in the density of the glass and also alter its refractive index. For instance, the use of a synthetic quartz glass ingot having an internal transmittance of less than 99.70% as a lens material for an exposure system which employs an ArF excimer laser as the light source may give rise to undesirable effects such as distortion of the image plane (or field curvature) due to changes in the refractive index of the lens material.

[0040] Thus, the burner must be configured and arranged as described above. For optimum operation of the burner, the silica-forming compound and oxygen are fed to the burner in such a mixing ratio that the molar amount of the silica-forming compound is at least 1.3 times, especially 2.0 to 3.0 times, the stoichiometry of oxygen.

[0041] Additionally, the molar ratio of the amount of actually fed oxygen to the stoichiometry of oxygen needed for the silica-forming compound (silane or siloxane compound) and hydrogen fed to the burner is preferably in a range of 0.6 to 1.3, more preferably 0.7 to 0.9.

[0042] Specifically, the silica-forming compound is fed to the center tube at a flow rate of preferably 0.3 to 0.7 Nm^3/hr, more preferably 0.4 to 0.5 Nm^3/hr. It is recommended that the combustion-supporting gas such as oxygen be fed to the

center tube at a flow rate of 2.0 to 4.0 Nm^3/hr, more preferably 2.5 to 3.5 Nm^3/hr. Additionally, an inert gas such as argon may be fed.

[0043] Preferred settings for the remaining tubes are such that the flow rate of combustion-supporting gas through the first enclosure tube is 0.3 to 2.5 Nm^3/hr; the flow rate of combustible gas through the second enclosure tube is 12 to 15 Nm^3/hr; the flow rate of combustible gas through the shell is 20 to 25 Nm^3/hr; the total flow rate of combustion-supporting gas through the nozzles is 10 to 16 Nm^3/hr; and the flow rate of combustion-supporting gas through the double-tube assembly is 2 to 5 Nm^3/hr. The gas flow velocity through the double-tube assembly is preferably in the range of 0.5 to 1.3 m/sec.

[0044] Once the necessary gases are fed to the burner as specified above and burnt to form an oxyhydrogen flame, the silica-forming compound undergoes vapor phase hydrolysis or oxidative decomposition to form silica fines which deposit on the target. The silica fines are simultaneously melted and vitrified. The vitrifying temperature has a distribution on the growth face. By setting a minimum temperature at this time to at least 1800°C, preferably at least 2000°C (with an upper limit of up to 2500°C, preferably up to 2400°C), the region of synthetic quartz glass which has an internal transmittance at wavelength 193.4 nm of at least 99.70% can be enlarged. The use of the inventive burner and the setting of an optimum gas balance such as that between oxygen and hydrogen greatly contribute to the melting and vitrifying temperature at the growth face.

[0045] With respect to the transmittance versus the melting and vitrifying temperature at the growth face, the inventors have discovered that the melting face temperature exerts an influence on the transmittance at wavelengths shorter than 200 nm, especially at the wavelength of ArF excimer laser light (193.4 nm). Thus, a higher melting and vitrifying temperature makes it possible to maintain an internal transmittance of at least 99.70%. Moreover, within this range of conditions, it is possible to maintain the hydrogen molecule concentration in the synthetic quartz glass at a level of at least 3×10^{18} molecules/cm^3 and thus achieve a long-term stability (sufficient to restrain the transmittance from lowering) during excimer laser irradiation. It also becomes possible to produce a jumbo synthetic quartz glass ingot having a diameter of at least 150 mm, typically 150 to 500 mm, and especially 200 to 300 mm.

[0046] It is preferred for the synthetic quartz glass ingot produced using the inventive burner to have an internal transmittance at wavelength 193.4 nm of at least 99.70% over the entire surface of a slice when the ingot is sliced perpendicular to its axis. Also preferably the glass has an OH group content of 500 to 1,300 ppm, especially 800 to 900 ppm. Moreover, a hydrogen molecule concentration of at least 3×10^{18} molecules/cm^3, preferably 3×10^{18} to 6×10^{18} molecules/cm^3, most preferably 3×10^{18} to 5×10^{18} molecules/cm^3 is desirable for good resistance to laser damage. Using the inventive burner, a synthetic quartz glass ingot having a large diameter can be produced, from which members for liquid crystal-related large-size glass substrates are effectively produced.

[0047] After a synthetic quartz glass ingot is produced, it is processed as by cylindrical grinding, thermoformed into a rectangular block as a mask substrate by heat melting at a temperature in the range of 1700 to 1800°C, annealed at a temperature in the range of 1000 to 1300°C for strain relief, sliced and polished, completing a synthetic quartz glass substrate. When the synthetic quartz glass ingot is used as optical lens material, it is subjected to homogenizing treatment, obtaining synthetic quartz glass free of striae in three directions. Specifically, both ends of a synthetic quartz glass ingot are welded to synthetic quartz glass supporting rods held in a lathe and the ingot is drawn out to a diameter of 80 mm. One end of the ingot is then strongly heated with an oxyhydrogen burner to at least 1,700°C, and preferably at least 1,800°C, so as to form a molten zone. Then, the opposed chucks are rotated at different speeds to apply shear stress to the molten zone, thereby homogenizing the quartz glass ingot. At the same time, the burner is moved from one end of the ingot to the other end so as to homogenize the hydroxyl group concentration and hydrogen concentration within the ingot growth face (homogenization by the zone melting method). The resulting synthetic quartz glass is typically shaped to the desired dimensions, and then preferably annealed for the glass to take a uniform fictive temperature (FT). Annealing can be carried out by a conventional method.

[0048] Synthetic quartz glass members thus obtained are useful as optical quartz glass members including excimer laser lenses, synthetic quartz glass substrates for photomasks, stepper illumination system lenses, and projection optical system lenses, windows, mirrors, beam splitters and prisms in the LSI field; and members for large-size quartz glass substrates such as color filter substrates, dust-proof glass substrates and facing glass substrates in the liquid crystal field for LCD.

EXAMPLE

[0049] The following examples are provided to illustrate the invention, and are not intended to limit the scope thereof. It is noted that in Examples, an internal transmittance was measured by ultraviolet spectrophotometry (Cary 400 by Varian Corp.).

Example and Comparative Example

[0050] A synthetic quartz glass ingot was produced by feeding methyltrimethoxysilane as the starting material to an inventive burner (FIG. 1) or a prior art burner (FIG. 3), effecting oxidative or combustion decomposition of the silane in an oxyhydrogen flame to form fine particles of silica, then depositing the silica particles on a rotating quartz target while melting and vitrifying them at the same time.

[0051] Specifically, as shown in FIG. 4, a quartz glass target 12 was mounted on a rotating heat-resistant support 11. Argon gas 15 was introduced into the methyltrimethoxysilane 14 held in a starting material vaporizer 13. Methyltrimethoxysilane 14 vapor was carried out of the vaporizer by the argon gas 15, and oxygen gas 16 was added to the silane-laden argon to form a gas mixture, which was then fed to the center tube of a main burner 17.

As shown in FIGS. 1 and 3, the main burner 17 was also fed the following gases, in outward order from the foregoing gas mixture at the center: oxygen gas 18, hydrogen gas 19, hydrogen gas 20, oxygen gas 21, and oxygen gas 22. The starting material, methyltrimethoxysilane 14 and an oxyhydrogen flame 23 were discharged from the main burner 17 toward the target 12. Fine particles of silica 24 were deposited on the target 12 and simultaneously melted and vitrified as clear glass, forming a synthetic quartz glass ingot 25. The ingot thus obtained had a diameter of 150 mm and a length of 500 mm. The flow velocity of combustion-supporting gas through the double-tube assembly was 0.6 m/sec. The parameters of the burners of Example and Comparative Example including the cross-sectional areas of tubes or nozzles, their ratio and gas feed rates are shown in Table 1.

Table 1

		Example (FIG. 1)		Comparative Example (FIG. 3)	
	Gas	Cross-sectional area (mm^2)	Gas flow rate (Nm^3/hr)	Cross-sectional area (mm^2)	Gas flow rate (Nm^3/hr)
Center tube	Silane	15	0.4	13	0.4
	O_2		3.0		2.0
	Ar		0.1		0.1
1st enclosure tube	O_2	30	1.0	32	1.0
2nd enclosure tube	H_2	50	14.0	60	15.0
Shell	H_2	1,700	24.0	1,800	25.0
Nozzles	O_2	150	12.0	80	16.0
Double-tube assembly	O_2	1,090	2.5	-	
Cross-sectional area ratio (%)	nozzles	8.8		4.4	
Note: Cross-sectional area ratio is a percentage of the total cross-sectional area of lumens of nozzles divided by the cross-sectional area of an annular space (prior to nozzle arrangement) between the second enclosure tube and the tubular shell.					

[0052] Next, the synthetic quartz glass ingots produced in Example and Comparative Example were sliced. Each slice was mirror finished. The distribution of initial transmittance at 193.4 nm of the slice from the center to the periphery was measured by ultraviolet spectrophotometry (Cary 400 by Varian Corp.). The results are shown in FIG. 5.

Claims

1. A burner for use in the manufacture of synthetic quartz glass, comprising a main burner (7) having a forwardly-directed front opening and a double-tube assembly (8) surrounding the front opening of the main burner (7);
a main burner (7) comprising an outer tubular shell (5) surrounding a multi-tube assembly (1) of three or more nested tubes (2,3,4), to define a gas discharge region for feeding a combustible gas in use around the multi-tube assembly (1) and inside the tubular shell (5), a plurality of nozzles (6) for feeding combustion-supporting gas in use being distributed in said gas discharge region, and wherein the multi-tube assembly (1) includes a centre tube (2) for feeding a silica-forming compound, a first enclosure tube (3) surrounding the centre tube (2) for feeding a combustion-

supporting gas and a second enclosure tube (4) surrounding the first enclosure tube (3) for feeding a combustible gas; the double-tube assembly (8) being spaced outwardly from the tubular shell (5) of the main burner (7) and comprising an outer tube (9) and an inner tube (10) disposed within the outer tube (9), defining between said outer and inner tubes (9,10) a passage for feeding combustion-supporting gas, the outer tube (9) surrounding and projecting forward relative to the front opening of the main burner (7), whereas the inner tube (10) has its front end axially in register with or behind the front opening of the main burner (7).

2. A burner according to claim 1 in which the total cross-sectional area of the gas discharge ports of the plural nozzles (6) distributed in said gas discharge region accounts for from 5% to 20% of the cross-sectional area of the gas discharge region between the multi-tube assembly (1) and the tubular shell (5).

3. A burner according to claim 1 or claim 2 in which the tubes (2,3,4) of the multi-tube assembly (1), the tubular shell (5) and the outer and inner tubes (9,10) of the double-tube assembly (8) are all circular in section and concentric.

4. A burner according to any one of the preceding claims in which the double-tube assembly (8) is made of quartz.

5. A method of making a synthetic quartz glass ingot using a burner as defined in any one of claims 1 to 4, comprising:

placing the burner to face a quartz glass target (12) mounted on a rotating support (11);
feeding silica-forming compound to the centre tube (2) of the burner, combustion-supporting gas to the first enclosure tube (3) and to the nozzle (6), combustible gas to the second enclosure tube (4) and to the gas discharge region inside the tubular shell (5), and combustion-supporting gas to said passage of the double-tube assembly (8);
forming an oxyhydrogen flame by combustion of the combustible gas in the combustion-supporting gas, thereby subjecting the silica-forming to vapour phase hydrolysis or oxidative decomposition to form silica fines;
depositing the silica fines on the target (12), with simultaneous melting and vitrification of the deposited silica fines to form quartz glass.

6. A method according to claim 5 wherein the silica-forming compound is a silane or siloxane, the combustion-supporting gas is oxygen and the combustible gas is hydrogen;
the silica-forming compound and oxygen are fed to the burner such that the molar amount of the silica-forming compound is at least 1.3 times the stoichiometric amount relative to the oxygen, and
the molar ratio, of the amount of oxygen actually fed to the stoichiometric amount of oxygen needed for the silica-forming compound and hydrogen fed to the burner, is from 0.6 to 1.3.

7. A method according to claim 5 or 6 wherein the combustion-supporting gas is fed through the double-tube assembly (8) at a flow velocity of from 0.5 to 1.3 m/sec.

8. A method according to any one of claims 5 to 7 in which
the flow rate of silica-forming compound to the centre tube (2) is from 0.3 to 0.7 Nm^3/hr;
the flow rate of combustion-supporting gas through the first enclosure tube (3) is from 0.3 to 2.5 Nm^3/hr;
the flow rate of combustible gas through the second enclosure tube (4) is from 12 to 15 Nm^3/hr;
the flow rate of combustible gas through the gas discharge region of the tubular shell (5) is from 20 to 25 Nm^3/hr;
the total flow of combustion-supporting gas through the nozzles (6) is from 10 to 16 Nm^3/hr;
the flow rate of combustion-supporting gas through the double-tube assembly (8) is from 2 to 5 Nm^3/hr.

9. A method according to any one of claims 5 to 8 wherein the resulting ingot has a diameter of at least 150 mm.

10. A method according to claim 9 wherein the resulting quartz ingot has a diameter of from 200 to 300 mm.

11. A method according to any one of claims 5 to 10, followed by slicing and polishing the synthetic quartz glass ingot to produce a synthetic quartz glass optical member.

Patentansprüche

1. Brenner zur Verwendung zur Herstellung von synthetischem Quarzglas, wobei dieser einen Hauptbrenner (7) mit einer nach vorne gerichteten, vorderen Öffnung und einer Doppelrohranordnung (8), die die vordere Öffnung des

Hauptbrenners (7) umgibt, umfasst;
wobei der Hauptbrenner (7) eine äußere röhrenförmige Hülle (5) umfasst, die eine Anordnung mit mehreren Rohren (1) mit drei oder mehreren ineinander geschachtelten Rohren (2, 3, 4) umgibt, um einen Gasausgabebereich zu definieren, um ein brennbares Gas bei Verwendung um die Anordnung mit mehreren Rohren (1) herum- und im Inneren der röhrenförmigen Hülle (5) zuzuführen, wobei eine Vielzahl von Düsen (6), um bei Verwendung ein die Verbrennung unterstützendes Gas zuzuführen, in dem Gasausgabebereich verteilt angeordnet ist und wobei die Anordnung mit mehreren Rohren (1) Folgendes umfasst: ein zentrales Rohr (2), um eine Siliciumdioxid bildende Verbindung zuzuführen; ein erstes Einfassungsrohr (3), das das zentrale Rohr (2) umgibt, um ein die Verbrennung unterstützendes Gas zuzuführen, und ein zweites Einfassungsrohr (4), das das erste Einfassungsrohr (3) umgibt, um ein brennbares Gas zuzuführen;
wobei die Doppelrohranordnung (8) nach außen von der röhrenförmigen Hülle (5) des Hauptbrenners (7) beabstandet ist und ein äußeres Rohr (9) und ein in dem äußeren Rohr (9) angeordnetes, inneres Rohr (10) umfasst, wobei zwischen dem äußeren und dem inneren Rohr (9, 10) ein Durchlass definiert ist, um ein die Verbrennung unterstützendes Gas zuzuführen, wobei das äußere Rohr (9) die vordere Öffnung des Hauptbrenners (7) umgibt und in Bezug auf diese vorsteht, während das vordere Ende des inneren Rohrs (10) axial fluchtend mit der vorderen Öffnung des Hauptbrenners (7) oder hinter dieser liegt.

2. Brenner nach Anspruch 1, worin die gesamte Querschnittsfläche der Gasausgabeöffnungen der Vielzahl in dem Gasausgabebereich verteilten Düsen (6) 5 bis 20 % der Querschnittsfläche des Gasausgabebereichs zwischen der Anordnung mit mehreren Rohren (1) und der röhrenförmigen Hülle (5) ausmacht.

3. Brenner nach Anspruch 1 oder 2, worin die Rohre (2, 3, 4) der Anordnung mit mehreren Rohren (1), die röhrenförmige Hülle (5) und das äußere und das innere Rohr (9, 10) der Doppelrohranordnung (8) alle einen kreisförmigen Querschnitt aufweisen und konzentrisch sind.

4. Brenner nach einem der vorangegangenen Ansprüche, in dem die Doppelrohranordnung (8) aus Quarz besteht.

5. Verfahren zur Herstellung eines synthetischen Quarzglasrohlings unter Einsatz eines Brenners nach einem der Ansprüche 1 bis 4, wobei das Verfahren Folgendes umfasst:

das Platzieren eines Brenners, so dass dieser einem auf einem rotierenden Träger befestigten Quarzglas-Target (12) gegenüberliegend angeordnet ist;
das Zuführen einer Siliciumdioxid bildenden Verbindung in das zentrale Rohr (2) des Brenners, von die Verbrennung unterstützendem Gas in das erste Einfassungsrohr (3) und die Düse (6), von brennbarem Gas in das zweite Einfassungsrohr (4) und zu dem Gasausgabebereich im Inneren der röhrenförmigen Hülle (5) und von die Verbrennung unterstützendem Gas in den Durchlass der Doppelrohranordnung (8);
das Ausbilden einer Knallgasflamme durch die Verbrennung des brennbaren Gases in dem die Verbrennung unterstützenden Gas, wodurch die Siliciumdioxid bildende Verbindung zur Ausbildung von Siliciumdioxidfeingut einer Dampfphasenhydrolyse oder einer oxidativen Zersetzung unterzogen wird;
das Abscheiden des Siliciumdioxidfeinguts auf dem Target (12) bei gleichzeitigem Schmelzen und Verglasen des abgeschiedenen Siliciumdioxidfeinguts zur Ausbildung von Quarzglas.

6. Verfahren nach Anspruch 5, worin die Siliciumdioxid bildende Verbindung ein Silan oder Siloxan ist, wobei das die Verbrennung unterstützende Gas Sauerstoff ist und das brennbare Gas Wasserstoff ist;
die Siliciumdioxid bildende Verbindung und Sauerstoff dem Brenner so zugeführt werden, dass die Molmenge der Siliciumdioxid bildenden Verbindung zumindest dem 1,3-Fachen der stöchiometrischen Menge in Bezug auf den Sauerstoff entspricht, und
das Molverhältnis der tatsächlich zugeführten Sauerstoffmenge zu der stöchiometrischen Sauerstoffmenge, die für die Siliciumdioxid bildende Verbindung und den Wasserstoff, die dem Brenner zugeführt werden, erforderlich ist, 0,6 bis 1,3 beträgt.

7. Verfahren nach Anspruch 5 oder 6, worin das die Verbrennung unterstützende Gas durch die Doppelrohranordnung (8) mit einer Strömungsgeschwindigkeit von 0,5 bis 1,3 m/s zugeführt wird.

8. Verfahren nach einem der Ansprüche 5 bis 7, in dem die Strömungsrate der Siliciumdioxid bildenden Verbindung zu dem zentralen Rohr (2) 0,3 bis 0,7 Nm^3/h beträgt;
die Strömungsrate des die Verbrennung unterstützenden Gases durch das erste Einfassungsrohr (3) 0,3 bis 2,5 Nm^3/h beträgt;

die Strömungsrate des brennbaren Gases durch das zweite Einfassungsrohr (4) 12 bis 15 Nm^3/h beträgt;
die Strömungsrate des brennbaren Gases durch den Gasabgabebereich der röhrenförmigen Hülle (5) 20 bis 25 Nm^3/h beträgt;
die Gesamtströmungsrate des die Verbrennung unterstützenden Gases durch die Düsen (6) 10 bis 16 Nm^3/h beträgt und
die Strömungsrate des die Verbrennung unterstützenden Gases durch die Doppelrohranordnung (8) 2 bis 5 Nm^3/h beträgt.

9. Verfahren nach einem der Ansprüche 5 bis 8, worin der resultierende Rohling einen Durchmesser von zumindest 150 mm aufweist.

10. Verfahren nach Anspruch 9, worin der resultierende Quarzrohling einen Durchmesser von 200 bis 300 mm aufweist.

11. Verfahren nach einem der Ansprüche 5 bis 10, gefolgt davon, dass der synthetische Quarzglasrohling in Scheiben geschnitten und poliert wird, um ein optisches Element aus synthetischem Quarzglas herzustellen.

Revendications

1. Brûleur à utiliser dans la fabrication de verre de quartz synthétique, comprenant un brûleur principal (7) comportant une ouverture frontale dirigée vers l'avant et un ensemble double tube (8) entourant l'ouverture frontale du brûleur principal (7) ;
un brûleur principal (7) comprenant une coque tubulaire externe (5) entourant un ensemble multitube (1) de trois tubes emboîtés ou plus (2, 3, 4) pour définir une région d'évacuation des gaz pour fournir un gaz combustible en utilisation autour de l'ensemble multitube (1) et à l'intérieur de la coque tubulaire (5), une pluralité de buses (6) pour fournir un gaz de support de combustion en utilisation qui est distribué dans ladite région d'évacuation des gaz, et où l'ensemble multitube (1) inclut un tube central (2) pour fournir un composé formant de la silice, un premier tube formant enceinte (3) entourant le tube central (2) pour fournir un gaz de support de combustion et un second tube formant enceinte (4) entourant le premier tube formant enceinte (3) pour fournir un gaz combustible ;
l'ensemble double tube (8) étant espacé vers l'extérieur à partir de la coque tubulaire (5) du brûleur principal (7) et comprenant un tube externe (9) et un tube interne (10) disposé dans le tube externe (9), définissant entre lesdits tubes externe et interne (9, 10), un passage pour fournir le gaz supportant la combustion, le tube externe (9) entourant, et faisant saillie vers l'avant par rapport à l'ouverture frontale du brûleur principal (7), tandis que le tube interne (10) a son extrémité frontale axialement en alignement avec ou derrière l'ouverture frontale du brûleur principal (7).

2. Brûleur selon la revendication 1, dans lequel l'aire en coupe totale des orifices d'évacuation des gaz de la pluralité de buses (6) distribuées dans ladite région d'évacuation des gaz représente de 5 % à 20 % de l'aire en coupe de la région d'évacuation des gaz entre l'ensemble multitube (1) et la coque tubulaire (5).

3. Brûleur selon la revendication 1 ou la revendication 2, dans lequel les tubes (2, 3, 4) de l'ensemble multitube (1), la coque tubulaire (5) et les tubes externe et interne (9, 10) de l'ensemble double tube (8) sont tous circulaires en section et concentriques.

4. Brûleur selon l'une quelconque des revendications précédentes, dans lequel l'ensemble double tube (8) est constitué de quartz.

5. Procédé de fabrication d'un lingot de verre de quartz de synthèse utilisant un brûleur tel que défini dans l'une quelconque des revendications 1 à 4, comprenant les étapes consistant à :

placer le brûleur en face d'une cible de verre de quartz (12) montée sur un support rotatif (11) ;
fournir un composé formant de la silice au tube central (2) du brûleur, un gaz de support de combustion a premier tube formant enceinte (3) et à la buse (6), un gaz combustible au second tube formant enceinte (4) et à la région d'évacuation des gaz à l'intérieur de la coque tubulaire (5), et un gaz de support de combustion audit passage de l'ensemble double tube (8) ;
former une flamme oxyhydrique par combustion du gaz combustible dans le gaz de support de combustion, soumettant ainsi le composé formant de la silice à une hydrolyse en phase vapeur ou une décomposition oxydative pour former des fines de silice ;

déposer les fines de silice sur la cible (12), avec une fusion et une vitrification simultanées des fines de silice déposées pour former du verre de quartz.

6. Procédé selon la revendication 5, dans lequel le composé formant de la silice est un silane ou siloxane, le gaz de support de combustion est de l'oxygène et le gaz combustible est de l'hydrogène ;
le composé formant de la silice et l'oxygène sont fournis au brûleur de telle sorte que la quantité molaire du composé formant de la silice est au moins 1,3 fois la quantité stoechiométrique par rapport à l'oxygène, et
le rapport molaire, entre la quantité d'oxygène effectivement fournie et la quantité stoechiométrique d'oxygène nécessaire pour le composé formant de la silice et de l'hydrogène fournis au brûleur, est de 0,6 à 1,3.

7. Procédé selon la revendication 5 ou 6, dans lequel le gaz de support de combustion est fourni à travers l'ensemble double tube (8) à un débit de 0,5 à 1,3 m/s.

8. Procédé selon l'une quelconque des revendications 5 à 7, dans lequel le débit du composé formant de la silice au tube central (2) est de 0,3 à 0,7 Nm^3/h ;
le débit du gaz de support de combustion à travers le premier tube formant enceinte (3) est de 0,3 à 2,5 Nm^3/h ;
le débit du gaz combustible à travers le second tube formant enceinte (4) est de 12 à 15 Nm^3/h ;
le débit de gaz combustible à travers la région d'évacuation des gaz de la coque tubulaire (5) est de 20 à 25 Nm^3/h ;
le débit total du gaz de support de combustion à travers les buses (6) est de 10 à 16 Nm^3/h ;
le débit du gaz de support de combustion à travers l'ensemble double tube (8) est de 2 à 5 Nm^3/h

9. Procédé selon l'une quelconque des revendications 5 à 8, dans lequel le lingot résultant a un diamètre d'au moins 150 mm.

10. Procédé selon la revendication 9, dans lequel le lingot de quartz résultant a un diamètre de 200 à 300 mm.

11. Procédé selon l'une quelconque des revendications 5 à 10, suivi par un tranchage et un polissage du lingot de verre de quartz de synthèse pour produire un organe optique en verre de quartz de synthèse.

FIG.1

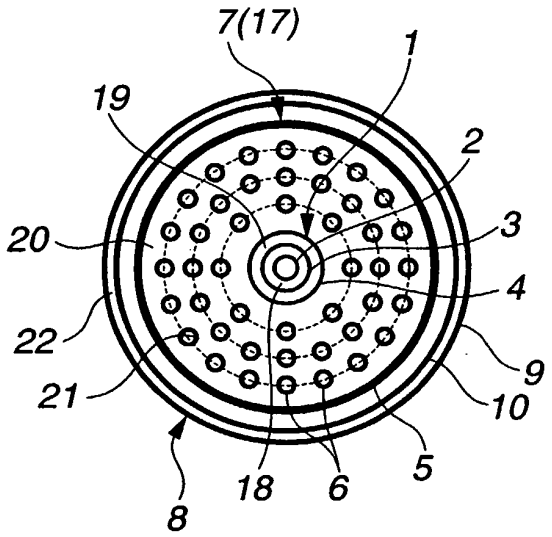


FIG.2

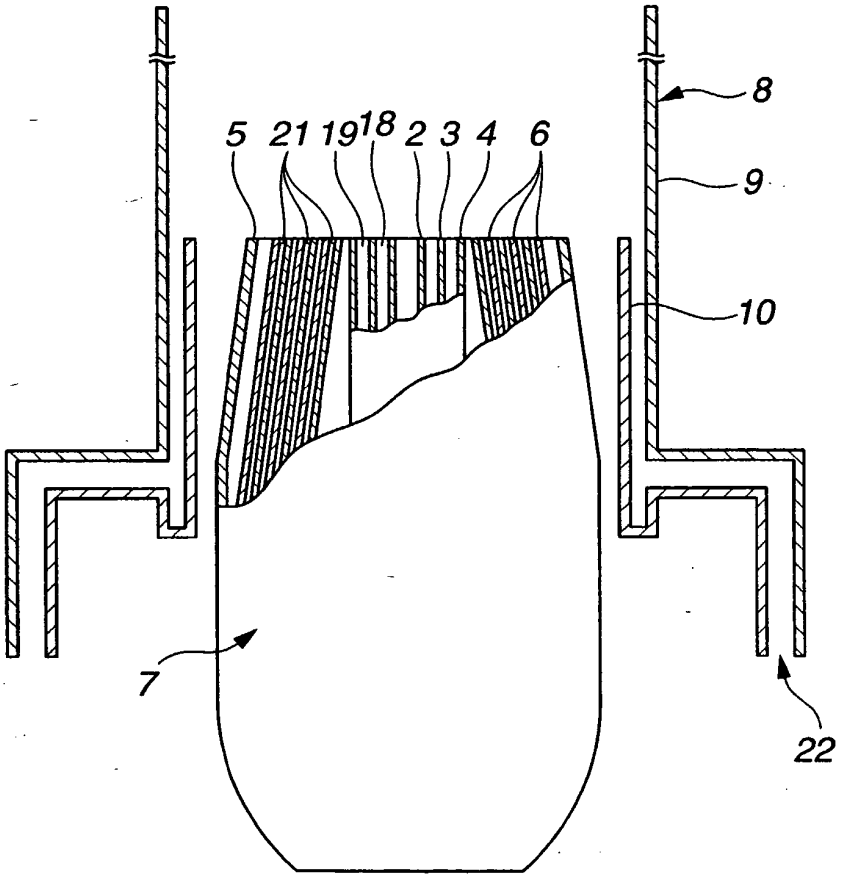


FIG.3
PRIOR ART

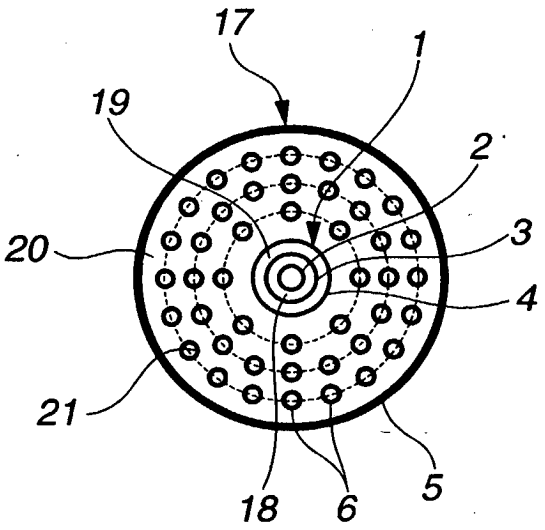


FIG.4

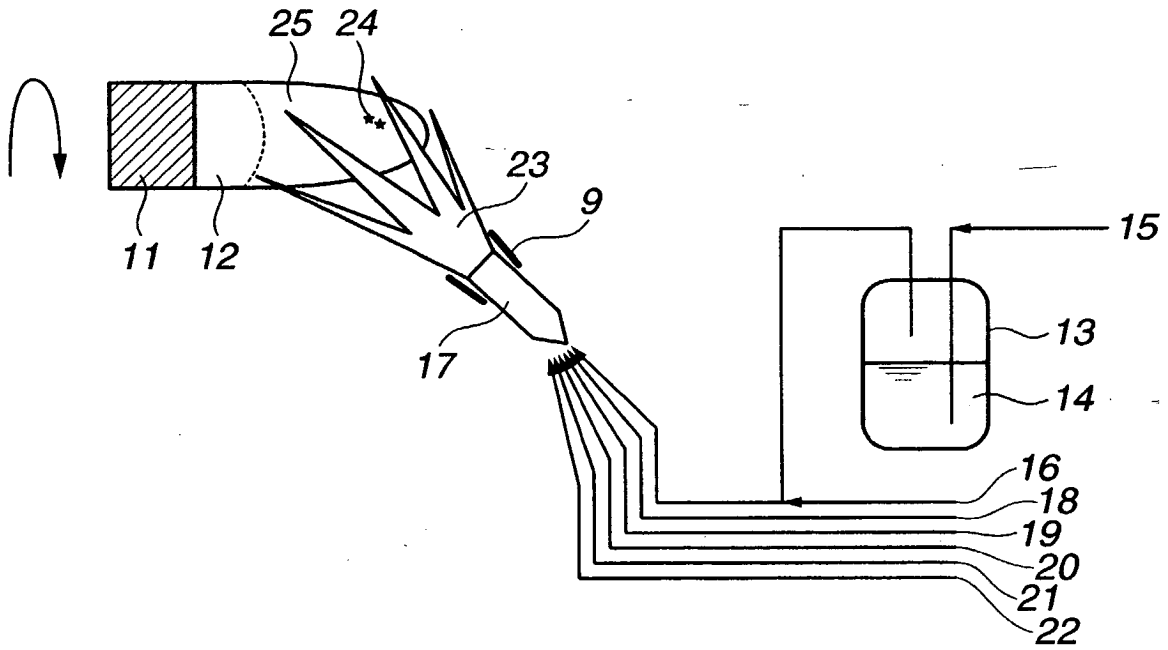
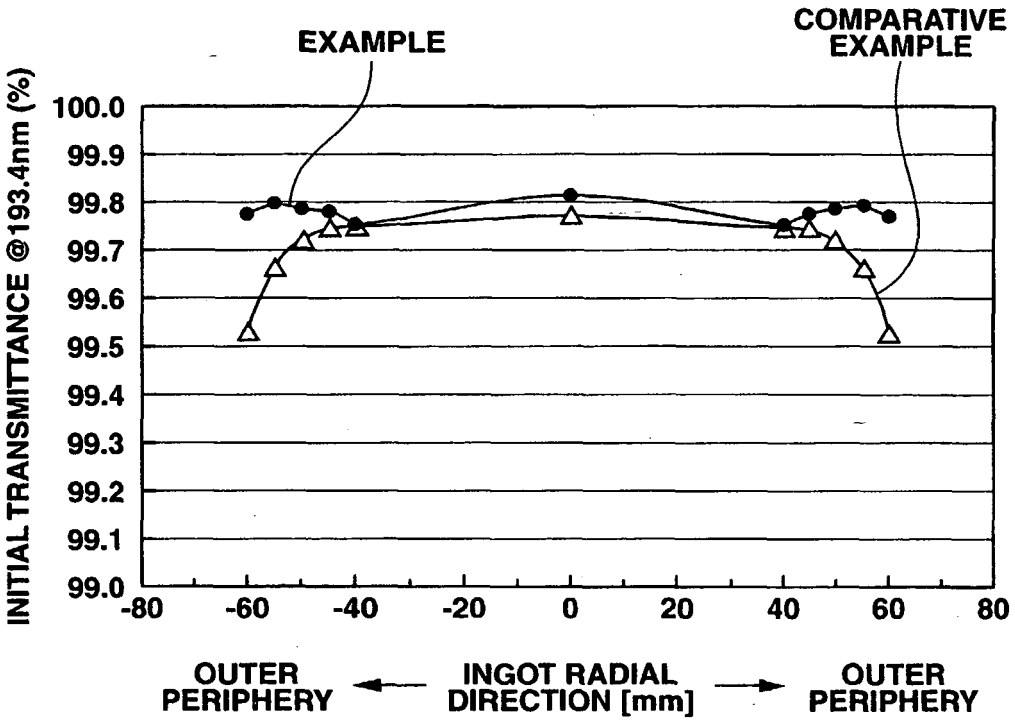


FIG.5


EXAMPLE
COMPARATIVE EXAMPLE
INITIAL TRANSMITTANCE @193.4nm (%)
100.0
99.9
99.8
99.7
99.6
99.5
99.4
99.3
99.2
99.1
99.0
-80
-60
-40
-20
0
20
40
60
80
OUTER PERIPHERY
INGOT RADIAL DIRECTION [mm]
OUTER PERIPHERY

REFERENCES CITED IN THE DESCRIPTION

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- JP 6305736 A **[0009]**
- EP 1278009 A **[0015]**
- EP 0943586 A **[0015]**
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